Docket No.: 21581-00445-US
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Tadashi Marumoto

Application No.: 10/580,916 Confirmation No.: 4796

Filed: August 18, 2006 Art Unit: 1794

For: INTERLAYER FILM FOR LAMINATE GLASS AND LAMINATED GLASS Examiner: R. G. A. Blackwell

DECLARATION UNDER 37 CFR 1.132

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Tadashi Marumoto, declare as follows:

1) I am an inventor of the present invention disclosed in the above referenced US patent application.

2) I received a Masters degree in material science from Nagoya University.

3) I have been employed by Sekisui Chemicals Co. Ltd., assignee of this application, since 2001, where I hold the position of Deputy Chief Engineer, with the responsibility for sub-managing.

4) The attached data was generated by my laboratory under my direction and/or supervision.

5) As shown by the results, those examples (Experimental Examples 3, 4 and 5) employing acetyl acetone in an amount of 0.008 to 0.1 parts by weight provide the excellent results according to the present invention. On the other hand, those examples

(Experimental Examples 1, 2, 5 and 6 in Experimental Result 1; and Experiment Result 2) resulted in formation of a colored spot in a metal coating on a glass surface and/or undesirable pummel value.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 2. Nov. 2009 By: Tadashi Marumoto

Tadashi Marumoto

Experimental Result 1

(Experimental Example 1)

(1) Synthesis of matrix resin

275 parts by weight of polyvinyl alcohol having an average polymerization degree of 1700 and a saponification degree of 99.2 mol% was added to 2890 parts by weight of pure water and the mixture was dissolved by heating. A temperature of this reaction system was adjusted to 15°C, and to this solution, 201 parts by weight of a 35 weight % hydrochloric acid and 157 parts by weight of n-butyl aldehyde were added, and a temperature of this mixture was kept at this temperature to precipitate a reactant. Then, the reaction system was kept at 60°C for 3 hours to complete the reaction. Then, the reaction system was cleaned with excessive water to wash out unreacted n-butyl aldehyde, and the hydrochloric acid catalyst was neutralized with an aqueous solution of sodium hydroxide, a general neutralizer, and further the reaction system was washed for 2 hours with excessive water and dried to obtain a polyvinyl butyral resin in white powder form. An average butyralization degree of this resin was 68.5 mol%.

(2) Water washing of matrix resin

The obtained polyvinyl butyral resin was washed by stirring for 12 hours in hot water. This operation was repeated three times. By adding magnesium heptanoate and potassium nonanoate to this washed resin, the contents of magnesium and alkali metal were adjusted to 20 ppm and 45 ppm, respectively. An amount of each metal was measured by ICP emission spectrometry.

(3) Production of interlayer film for a laminated glass

To 100 parts by weight of the polyvinyl butyral resin obtained, triethylene glycol bis(2-ethylhexanoate) as a liquid plasticizer and 0.5 parts by weight of acetyl acetone were added, and the mixture was kneaded with a plasto machine and then was extruded into sheet form from a mold with an extruder

to obtain an interlayer film for a laminated glass having a thickness of 745 μm.

(4) Production of laminated glass

The obtained interlayer film for a laminated glass was sandwiched between two float glasses (30 cm long by 30 cm wide by 2.5 mm thick) provided with a metal coating layer from its both ends, and this was put in a rubber bag and deaerated at a vacuum of 2.6 kPa for 20 minutes, and then this was moved into an oven in a state of being deaerated and subjected to vacuum press while being further retained at 90°C for 30 minutes. A laminated glass formed preliminarily by thus attaching the float glasses to each other by applying pressure was subjected to attaching by pressure under the conditions of 135°C and a pressure of 1.2 MPa for 20 minutes in an autoclave to obtain a laminated glass.

(Experimental Example 2)

An interlayer film for a laminated glass and a laminated glass were produced by following the same procedure as in Experimental Example 1 except for not adding acetyl acetone 0.2 parts by weight.

(Experimental Example 3)

An interlayer film for a laminated glass and a laminated glass were produced by following the same procedure as in Experimental Example 1 except for adding acetyl acetone 0.1 parts by weight.

(Experimental Example 4)

An interlayer film for a laminated glass and a laminated glass were produced by following the same procedure as in Experimental Example 1 except for adding acetyl acetone 0.008 parts by weight.

(Experimental Example 5)

An interlayer film for a laminated glass and a laminated

glass were produced by following the same procedure as in Experimental Example 1 except for adding acetyl acetone 0.005 parts by weight.

(Experimental Example 6)

An interlayer film for a laminated glass and a laminated glass were produced by following the same procedure as in Experimental Example 1 except for not adding acetyl acetone.

<Evaluation>

On each laminated glass obtained in Experimental Example 1 to 6, the following evaluations were performed. The results of evaluations are shown in Table.

(1) Check of formation of colored spot in metal coating on glass surface

The obtained laminated glass was placed under the conditions of 50°C and a humidity of 97% for 24 hours and visually rated according to the following criteria.

o There was not the formation of a colored spot in a metal coating on a glass surface.

× There was the formation of a colored spot in a metal coating on a glass surface.

(2) Measurement of pummel value

A temperature of the obtained laminated glass was adjusted within a range of -18°C±0.6°C for 16 hours, and a center area (an area of 150 mm long by 150 mm wide) of this laminated glass was struck with a hammer having a head weight of 0.45 kg and the laminated glass was milled till a diameter of a particle of milled glass becomes 6 mm or less, and a percentage of an interlayer film exposed by partial peeling off of the glass was measured and a pummel value was determined from the above-mentioned Table 1. The pummel value was evaluated according to the following criteria.

o pummel value: 3 to 6

× pummel value: 0 to 2, and 7 to 8

	Mg Content (ppm)	Alkali Metal Content (ppm)	Acetyl Acetone (parts by weight)	Corrosion	Pummel Value
Experimental Example 1	20	45	0.5	×	×
Experimental Example 2	20	45	0.2	×	×
Experimental Example 3	20	45	0.1	○	○
Experimental Example 4	20	45	0.008	○	○
Experimental Example 5	20	45	0.005	○	×
Experimental Example 6	20	45	0	○	×

Experimental Result 2

(Experimental Example)

(1) Synthesis of poly(vinyl butyral) Resin

A reactor equipped with a stirring means was charged with 2900 weight parts of deionized water and 198 weight parts of a poly(vinyl alcohol) with an average polymerization degree of 1700 and a saponification degree of 99.2 mole % (corresponding to 4.5 mols of vinyl alcohol) and the charge was heated to 95.degree. C. with stirring for dissolving. After this solution was cooled to 30.degree. C., 208 weight parts (2.1 moles) of 35 weight % hydrochloric acid and 152 weight parts (2.1 moles) of n-butyraldehyde were added. After the liquid temperature was lowered to 2.degree. C., the reaction system was maintained at this temperature to precipitate the poly(vinyl butyral) resin. The liquid temperature was then raised to 30.degree. C. and maintained at this level for 5 hours. Thereafter, the reaction mixture was neutralized with 156 weight parts (1.8 moles) of sodium hydrogencarbonate, washed with water and dried to provide a poly(vinyl butyral) resin with a butyralization degree of 65 mole %.

The sodium content of this poly(vinyl butyral) resin was 50 ppm as determined by ICP emission spectrometry. The particle diameter of the sodium salt was 12 .mu.m.

(2) Production of a Resin Film

One hundred (100) weight parts of the poly(vinyl butyral) resin obtained as above, 40 weight parts of triethylene glycol di-2-ethylbutyrate, 0.3 weight part of acetylacetone, 0.04 weight part of magnesium 2-ethylbutyrate and 0.05 weight part of modified silicone oil were fed to a mixing roll and kneaded. Using a pressing machine, this kneaded material was press-molded at 150.degree. C. and 120 kg/cm.sup.2 for 30 minutes to provide a resin film of 0.8 mm in thickness.

(3) Production of laminated glass

The obtained interlayer film for a laminated glass was sandwiched between two float glasses (30 cm long by 30 cm wide by 2.5 mm thick) provided with a metal coating layer from its both ends, and this was put in a rubber bag and deaerated at a vacuum of 2.6 kPa for 20 minutes, and then this was moved into an oven in a state of being deaerated and subjected to vacuum press while being further retained at 90°C for 30 minutes. A laminated glass formed preliminarily by thus attaching the float glasses to each other by applying pressure was subjected to attaching by pressure under the conditions of 135°C and a pressure of 1.2 MPa for 20 minutes in an autoclave to obtain a laminated glass.

<Evaluation>

On each laminated glass obtained in Experimental Example, the following evaluations were performed. The results of evaluations are shown in Table.

(1) Check of formation of colored spot in metal coating on glass surface

The obtained laminated glass was placed under the conditions of 50°C and a humidity of 97% for 24 hours and visually rated according to the following criteria.

o There was not the formation of a colored spot in a metal coating on a glass surface.

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A temperature of the obtained laminated glass was adjusted within a range of -18°C±0.6°C for 16 hours, and a center area (an area of 150 mm long by 150 mm wide) of this laminated glass was struck with a hammer having a head weight of 0.45 kg and the laminated glass was milled till a diameter of a particle of milled glass becomes 6 mm or less, and a percentage of an interlayer film exposed by partial peeling off of the glass was measured and a pummel value was determined from the above-mentioned Table 1. The pummel value

was evaluated according to the following criteria.

o pummel value: 3 to 6

× pummel value: 0 to 2, and 7 to 8

	Mg Content of resin film (ppm)	Sodium Content of PVB resin (ppm)	Acetyl Acetone (parts by weight)	Corrosion	Pummel Value
Experimental Example	27	50	0.3	x	x